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VAPOUR PHASE GROWTH OF THICK MONOCRYSTALLINE GaN EPITAXIAL LAYERS BY SANDWICH-METHOD.

Interim Technical Report

by

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13. ABSTRACT

Comparison studies of the crystal quality of SiC substrates grown by Lely method and sublimation sandwich-method (SSM) was conducted. SSM-substrates was shown to have considerably promise for high quality GaN epilayers growth. Conditions of the surface treatment of the SiC substrates were optimized for GaN epilayers growth. The best results were reached on very close to (0001) Si- substrate surfaces etched by sublimation technique. Monocrystalline GaN layers was shown to may be grown on the SiC substrates without any buffer layer at temperatures 1150-1200° C. Very high growth rates of the GaN epilayers up to 1 mm/h is possible. In result heteroepitaxial layers of the large area (more than 10 mm^2) and thickness up to 200 microns were grown by SSM.

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REPORT

SiC substrates grown by Lely method and sublimation sandwich-method (SSM) were studied by x-ray diffractometry method. The full width at half maximum (FWHM) for SSM substrate was found to be from 15 to 24 arc. sec at (0006) rocking curve that was close to meaning of FWHM (about 13 arc.sec.) for perfect SiC Lely crystals. It testifies that SSM-substrates have considerably promise for high quality GaN epilayers.

There were proposed normalized method of characterization based on usual x-ray rocking curves (RC) and small-angle scattering of x-ray (S-AS) method . The broadening of RC and the shape of S-AS spectrum revealed treatment quality of the surface, domain size (50 to 500 Angstroms) and their misorientation from 5 to 15 arc.sec..

Conditions of the surface treatment of the SiC substrates were optimized for GaN epilayers growth. The best results were reached in case of very close to (0001) Si- substrate surfaces etched by sublimation technique.

Growth kinetic and surface morphology of GaN epilayers was studied. Effect of substrate surface orientation and growth condition on the structural quality of the epilayers were discovered. GaN layers was shown to may be grown on the SiC substrates without any buffer layer at temperatures 1150-1200° C. Very high growth rate up to 1 mm/h was shown to be reached on SiC substrates. Heteroepitaxial layers of the large area (more than 10 mm^2) and thickness up to 200 microns were grown. Altogether we received 24 epitaxial structures of GaN on SiC substrates, grown both by Lely and SSM method.

We used modifyed small-angle scattering of X-rays for measurement (estimation at first stage) domain size of GaN crystals. Usual broadening rocking curve shows formation of misoriented sites in researched volume.

Luminescent properties of GaN epilayers were studied by color cathodoluminescence scanning electron microscopy. Influence of structural defects and deformation fields on the luminescence spectrum is revealed.

The results of research are submitted on the international conferences - 4 reports.

List of reports

1. M.E.Boiko, E.N. Mokhov, Yu.G. Shreter,. "Small-angle scattering of X-rays (S-AS) of GaN, deposited on SiC and Al_2O_3 substrates. E-MRS 97 Strasbourg ICAM`97 MRS-Strasbourg June 16-20, 1997.

2. M.E.Boiko Studying Crystal Domains with Small-Angle Scattering".Moscow-Dubna 26-27 May 1997. National Conference on Application of X-ray, Synchrotron Radiation, Neutron and Electron for Material Characterization (RSNE-97) Moscow-Dubna 26-27 May 1997.

3. Michael E. Boiko Incommensurate domains in gallium nitride plain slabs. The second International Conference on LOW DIMENSIONAL STRUCTURES AND DEVICES LDSD '97, Lisbon, Portugal 19-21 May 1997

4.E.N.Mokhov, A.D.Roenkov, G.V.Saparin, S.K. Obyden, P.V.Ivannikov and J.Freitas Characterization of GaN epitaxial Layers by Color SEM-CL Method. Scanning - 97,Monterey {USA), Apr.18-22 1997

(2) Brief description of the research plans on staying period

1. Optimization of modes of growth of thick layers with the large area of a surface:
   - of temperature modes;
   - of geometry of modes systems;
   - of conditions of submission of gas
2. Characterization of GaN thick layers:
- Structural perfection (x-ray method);
- optical, electrical and luminescent measurements;
- electron paramagnetic resonance study.

1.Optimization of Substrate Surface Treatment before GaN Layer Deposition.

1.1 SiC Substrate Growth and Characterization

The substrates to be studied were SiC crystals, mostly 6H, differing in the growth conditions. Group-I substrates consisted of Lely crystals grown at a temperature close to 2550° C [1]. Such crystals grow as wafers with at least one well-defined habit (0001) face. This crystal shape facilitates the production of substrates with a singular surface having a very small misorientation angle. The crystals of this group possess a fairly high structural perfection, as is evidenced by X-ray analysis [2]. The dislocation density varies between 10^2 - 10^3 cm^{-2} , and the density of micropipes and pores is usually below 10^1 cm^{-2}. However, the area of a Lely crystal is relatively small, and the linear dimensions rarely exceed 1 cm. Besides, the shape of the crystals grown under identical conditions varies considerably, e.g. crystals are often stretched in one direction.

For this reason, much effort has been made to obtain SiC substrates by other methods, in particular, by sublimation growth consisting in the transport of SiC vapors from a hot polycrystalline source to a cooler single crystal substrate. Deposition at low pressures (< 0.1 atm) made it possible to reduce the growth temperature to 2100 - 2400° C. The first SiC crystals of 15 mm diameter were grown in this way by Tairov and Tsvetkov [3]. At present, bulk SiC crystals for substrate application are being produced in the USA, Germany, Japan, and Russia. The best commercial SiC ingots are made by the CREE firm (USA). For example, 6H -SiC and 4H -SiC ingots with a 2 inch diameter have been produced [4] for GaN epitaxial layer growth. The crystals are usually of n-type conductivity due to the presence of donor nitrogen, whose concentration varies within $(0.5 - 3)10^{18}$ cm^{-3} .

The growth techniques employed by various research groups do not differ much, though they bear different names. More often, the method of growing SiC bulk ingots is termed as a modified Lely technique.

Modified Lely SiC single crystals have been studied by several workers [2, 5], who have found that their structural perfection is still inferior to that of conventional Lely crystals. For example, they have much higher densities of dislocations (10^4 cm^{-2}) and micropipes (10^2 cm^{-2}) and often contain inclusions of other SiC polytypes. Also, there are misoriented and strained regions distributed nonuniformly over the wafer area. Such defects are readily detectable by X-ray diffractometry. They lead to much larger halfwidths of the reflection curves. SiC crystals of this kind were found to have a mosaic structure due to the co-existence of large and small structural units slightly misoriented relative to one another [5].

The presence of residual strains in SiC ingots, absent from Lely crystals, seem to be associated with the lower growth temperatures. Indeed, we have shown that relaxation of the strains introduced by mechanical damage of the crystal surface occurs at fairly high temperatures and is not completed even at 2300° C [6]. Note also that the growth temperatures of bulk crystals are insufficient for the annealing of nonequilibrium point defects, which may persist as clusters or associates detectable by various physical methods [7].

In the present work, we have used SiC substrates grown by sublimation sandwich-method (SSM). As early as 1970, we first demonstrated the possibility to grow high quality

SiC layers, using the SSM, by which we achieved high growth rates (> 1 mm/hour) at relatively low temperatures (about 1900° C) in vacuum (P ~ 10^{-5} Torr) [8]. Later, we employed the same technique for growing 6H.15R-SiC and 4H-SiC ingots of 1 inch diameter [9]. From these, we made SiC substrates to be used in the present work (Group-II substrates).

The structural perfection of the Lely and sublimation SiC substrates was tested by X-ray diffraction. A standard (omega) scan was used for the characterization of SiC substrates and GaN epilayers. The crystal monochromator in the X-ray diffractometer was a (0001) SiC Lely crystal. The full width at the half minimum (FWHM) of the crystal was less than 13 arc.sec for the (0006) rocking curve and 8 arc.sec for (00012). The high precision of the X-ray optics adjustment provided standard rocking curves for the characterization.

The FWHM measurements of the SiC substrates grown by different techniques are summarized in Table 1. On the whole, they indicate a high quality of the substrates produced by sandwich sublimation: they have been found to possess the same structural perfection as crystals grown by the modified Lely technique. There were proposed normalized method of characterization based on usual x-ray rocking curves (RC) and small-angle scattering of x-ray (S-AS) method. Each measurement were joined to another. The broadening of RC and the shape of S-AS spectrum revealed treatment quality of the surface, domain size (50 to 500 Angstroms) and their misorientation from 5 to 15 arc.sec..

In the Fig. 1. we see three rocking curves: (0006), (000 12) from the random part of the crystal and (0006) from the best part of the Lely crystal. It is well seen that (0006) monochromator is in good agreement with (0006) rocking curve. In common thinks it is forbidden to compare (006) and (000 12) rocking curve. In the special choosing we find the place with narrow RC close to theoretical data.

In the Fig.2 we see (0006) RC for SSM substrate. The wide RC corresponds to the surface of the plate after saw. The narrow RC corresponds to polished and etched surface. It is seen that the quality of Lely crystals coincide with SSM substrates.

1.2 Substrate Preparation for GaN Layer Growth

The SiC crystals used as substrates represented wafers of 450 - 500 μm thick with the base surfaces close to the (0001) plane. The substrates had a misorientation angle varying between 0 and 5° with respect to the (0001) face. The samples were subjected to pre-grinding and polishing with diamond pastes followed by etching in the alkali KOH melt for several minutes. The (0001) C face is known to etch fast, at a rate 20 - 100 times that of the (0001) Si face. The Si face, unlike the C face, is characterized by selective etching occurring mostly in the vicinity of structural defects—dislocations, scratches, second-phase inclusions, etc. This fact can serve for identification of the surface type, because micropipes, dislocations, pores, scratches, and other mechanical defects become decorated on the Si face. The dislocation density in the substrates of interest varied from 10^2 to 10^4 cm^{-2} and the micropipe density was 10^1 cm^{-2} or less.

The structure of the grind-damaged near-surface layer of the crystal is, according to [10], complex and includes an inner sublayer with residual strains. The total thickness of the damaged layer is 30 - 50 μm; it should be removed prior to the epilayer growth, because on heating above 1100° C the relaxation of residual strains occurs to form dislocations [11]. For this reason, we removed all the damaged layer before the growth.

With the (0001) C face, this was done by chemical etching in the KOH melt at 480° C for 30 min to remove a 50 μm layer. This procedure proved impossible for the polar (0001) Si surface because of its selective etching and a low etching rate (10^2 times less than for the (0001) C face). Ion-plasma etching is normally used in this case [12] but its rate is too low to remove all the damage defects from the subsurface layer.

To avoid these difficulties, we used a specially developed technique — sublimation etching [13] based on the same sublimation sandwich-method with the exception that the growth cell was placed in a thermal field with an opposite temperature gradient as compared to the growth gradient. In other words, the SiC wafer is at a higher temperature and evaporates during the heating. The sublimation etching rate varies not only with the temperature but also with the temperature gradient and the vapor composition [14]. In absence of impurities, the desired evaporation rates (about 1 μm/min) are attained at about 1800° C.

An important condition for quality etching is excessive Si vapor in the sublimation cell, since even a slight Si deficit results in partial graphitization of the surface to be evaporated, leading to its lower morphological quality. For this reason, the sublimation etching is carried out in a sandwich containing excessive silicon. A similar effect can be achieved by introducing tantalum [15] or some other impurities into the cell for gettering the excessive carbon. The influence of tantalum on the SiC sublimation mass transfer was partly studied earlier [16].

To optimize the treatment of the SiC substrate surface used for the epitaxial growth of GaN layers, we studied the influence, on the layer quality, of structural and morphological substrate defects, of a pre-deposited buffer layer, and of the way of removing the damaged layer, i.e. polishing, chemical or sublimation etching. The growth occurs on the polar substrate surfaces, (0001) C and (0001) Si, and on those misoriented at an angle of 0.5 and 5° C to them. The substrate thickness varied from 150 to 450 μm in some experiments.

The on-grown layer quality was controlled by a complex analysis of the layer parameters by optical and scanning electron microscopy, X-ray diffraction, and the luminescence technique.

2. Optimization of growth condition of GaN thick layers with large substrate surface area

2.1. GaN Epilayer Growth by Sublimation Sandwich- Method

The techniques commonly used at present for the production of GaN epilayers are metal organic chemical vapor deposition (MOCVD) and molecular beam epitaxy (MBE) [17]. They have provided quality layers of p- and n-types of conductivity. But a disadvantage of these methods is a very low growth rate, which normally varies from several fractions of a micrometer to several tens of micrometers per hour. We suggested another approach which we termed as sublimation sandwich-method (SSM) [18] providing much higher growth rates and a fairly high structural perfection of the on-grown layers [19].

The principle of this method is that the source of initial material, (a powder or a polycrystalline ingot) and a single crystal seed are placed parallel to each other, with a small gap between them, into the transverse temperature gradient at the growth temperature.

The main advantages of this arrangement are:

—effective mass transfer of material from the source to the substrate at a maximum growth rate of about 1 - 2 mm/hour;

—the creation of nearly quasi-equilibrium conditions inside the sandwich cell providing a homogeneous deposition of sublimating substances in a wide range of temperatures, temperature gradients, and ambient pressures;

—controllable variation of the vapor composition in the growth cell in order to obtain a material with a desired doping level and impurity composition or with a deviation from stoichiometry;

—a smaller effective cell volume, which reduces the consumption of the sublimating material (the percentage of transported material is close to 100%), increasing its purity as to residual impurities;

—control over the nucleation and growth of a desired crystal polytype, e.g. of high quality heteroepitaxial layers of SiC polytypes: 4H, 6H, 3C, and 15R;

—effective growth in the absence of surface-active impurities usually serving as transporters of sublimating material;

—easy creation of uniform thermal fields inside the sandwich as a means of improving the crystal quality;

—control of the intrinsic point defect ensemble in the on-grown samples owing to the wide temperature range used (900° C for SiC and 300° C for GaN), inaccessible to other methods;

—easier access to fundamental data on the growth process, possible simulation of the mass transfer processes during sublimation, and the model verification; this provides a better understanding of the crystal formation and elementary doping processes.

2.2 Experimental

GaN epilayers were grown in a horizontal quartz reactor in a microwave furnace (Figure 1). The growth cell consisted of a vapor source and a substrate separated by a narrow gap of 5 - 10 mm, placed into a temperature gradient zone. The source material was metallic Ga or GaN powder.

The crystal growth was performed in the temperature range of 1100 - 1300° C with the temperature gradient between the source and the substrate varying from 10° C to 50° C. Before the deposition of a GaN epilayer, the reactive zone was evacuated at 500° C. The growth was carried out in ammonia flow at atmospheric pressure, with the flow rate varying between 10 and 50 liters/hour.

2.3 Growth Kinetics

Single crystal GaN layers were grown in the temperature range 1050 - 1230° C. Note that the optimal growth temperature for such layers (1150 - 1200° C) is higher than in other growth techniques. This enabled the authors of work [20] to suggest another name—high temperature gas epitaxy—for their technique absolutely identical to sandwich sublimation. This temperature range provided the growth of homogeneous GaN layers, containing no second-phase inclusions, at very high rates (up to 1 mm/hour). Interestingly, under optimal growth conditions perfect GaN layers could be obtained without buffer pre-deposition.

Free Ga inclusions were observed only at temperatures above 1230° C; they were localized in the vicinity of structural and morphological defects. The epilayers grown under

these conditions usually had a high pore density. On the contrary, at temperatures below 1050° C, the probability of microcrystalline growth failure followed by formation of individual GaN crystals with a planar or prismatic shape became much higher. Then microcrystalline growth proved possible only on substrates with a pre-deposited GaN layer at maximum temperature gradients between the source and the substrate.

Figure 3 shows the temperature variation of the growth rate. One can see that it rises with temperature (up to 1200° C), reaching a value of 1 mm/hour. We would like to note that these are record rates for epitaxial deposition of single crystal GaN layers and several times higher than for hybrid gas epitaxy [17]. High deposition rates have also been achieved by other investigators using sandwich sublimation for GaN layer growth [20].

Further temperature increase leads to a sharp drop of the GaN deposition rate induced by thermal dissociation of the growing layer. This conclusion is supported by the appearance of Ga droplets on the surface.

Figure 3 shows the growth rate variation as a function of the gap size in the sandwich cell. It is seen that the rate decreases with the larger cell size, and the highest rates are achievable with small gaps where the growth process however becomes irreversible. One should bear in mind that as the gap size changes, not only does the Ga supply efficiency rises but the ammonia flow through the growth cell changes too, affecting the GaN growth rate. Obviously, the growth rate variation with ammonia flow has a complicated, nonmonotonic character. For example, the deposition rate of GaN decreases with low ammonia flows because of its dissociation. But the growth rate decreases even in large ammonia flows due to the removal of Ga molecules from the sandwich cell.

2.4 Structural and Morphological Defects in GaN Epilayers

The epilayers produced have been found to contain the following types of defects: cracks, second-phase inclusions, pores, dislocations, and surface undulations. We will discuss them individually.

Cracks

Cracks appeared in epilayers of 20 μm thick and more and were localized primarily near the substrate-epilayer boundary. It is interesting that the luminescence in the vicinity of cracks changed from short-wave (dark blue and violet) to yellow. The probability of crack formation abruptly decreased with decreasing rate of the substrate cooling. It seems clear, therefore, that their formation is associated with the differences in the thermal expansion coefficients.

We have also found the dependence of the crack density on the substrate thickness and misorientation angle relative to the (0001) face. The appropriate technological corrections have been introduced to minimize the probability of crack formation in GaN epilayers grown on SiC substrates.

Second-phase inclusions

Usually, second-phase inclusions represent liberated gallium and are observed in the vicinity of pores and other morphological defects. The inclusions are due to the active nitrogen deficiency near the growing surface, so a more powerful ammonia flow into the growth cell and a lowern growth temperature contribute to the elimination of such defects.

Pores and micropipes

These are the most characteristic defects in thick GaN epilayers. Their density strongly depends on the growth conditions and the state of the substrate surface. The pore density usually rises with increasing growth temperature or with decreasing ammonia flow. We suggest that porosity is associated with liberation of, say, gallium in the second phase

because of the deficit of active Ga. The pore formation is stimulated by a poor substrate surface treatment, e.g. incomplete removal of material after the damaged layer grinding, or the presence of individual morphological imperfections on this layer. Elevated pore density has also been observed in GaN epilayers deposited on substrate surfaces with a 1-5° misorientation relative to the (0001) face. What causes this effect is not quite clear yet.

Note that pores and micropipes present in the SiC substrate are inherited by the GaN epilayer, so the best epilayer samples were obtained on SiC substrates of high structural perfection.

Morphological defects

The morphology of GaN epilayer surfaces strongly depends on the substrate orientation. Relatively smooth layers grow on the (0001) Si face. Under the same conditions, GaN epilayers grown on (0001) C substrates have a rough surface covered by hexagonal growth undulations. At higher growth rates, individual prismatic crystals arise on the C face. We attribute the above defects to the fact that the growth process occurring on the carbon face involves three-dimensional seeds, while on the silicon face two-dimensional GaN seed are formed and then outgrow tangentially. We discussed this difference in the epitaxial growth mechanisms earlier [21] and interpreted it as being due to the specific chemical bonding of silicon and carbon atoms resulting in a higher surface energy of the (0001) Si face as compared to the (0001) C face.

Conclusion

The present study has demonstrated the possibility of growing single crystal GaN epilayers on SiC substrates at high rates, up to 1 mm/hour. The substrate quality has been found to affect significantly the epilayer perfection. The best GaN epilayers were grown on (0001) Si substrates with small misorientation angles by using sublimation etching. The growth of quality GaN epilayers on buffer-free substrates at temperatures of 1150 - 1200° C has been shown.

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Table 1

N	Method of growth	Polytype	Rocking curve	FWHM
1.	Lely	6H	0006	13"
2.	Lely	6H	0006	24"
3.	Lely	6H	0006	15"
4.	Lely	6H	0006	15"
5.	SSM	6H	0006	17"
6.	SSM	6H	0006	24
7.	SSM	6H	0006	22
8.	SSM	4H	0006	24"
9.	SSM	4H	0006	15
10.	SSM	4H	0006	16"

X-ray diffraction study of SiC substrates grown by different methods
Lely - free-standing Lely crystals
SSM - bulk crystals grown by crystals grown by sublimation sandwich - method

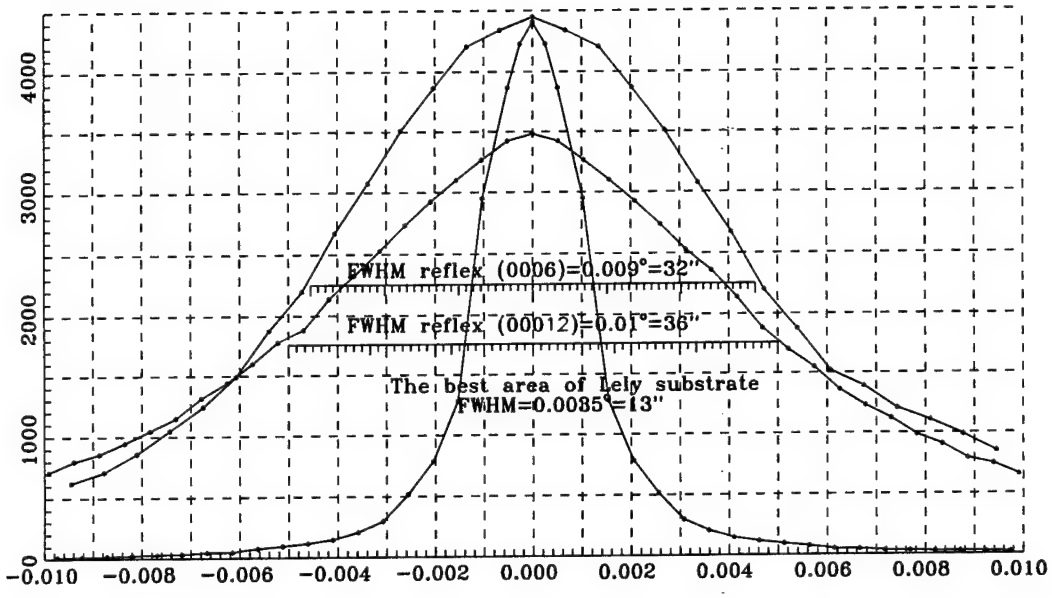

Fig.1. Usual rocking curves of Lely plates
(1116) SiC Wo=17.5arc deg
(000 12) Sic Wo =35arc.deg
(0006) SiC for the best part of Lely plate

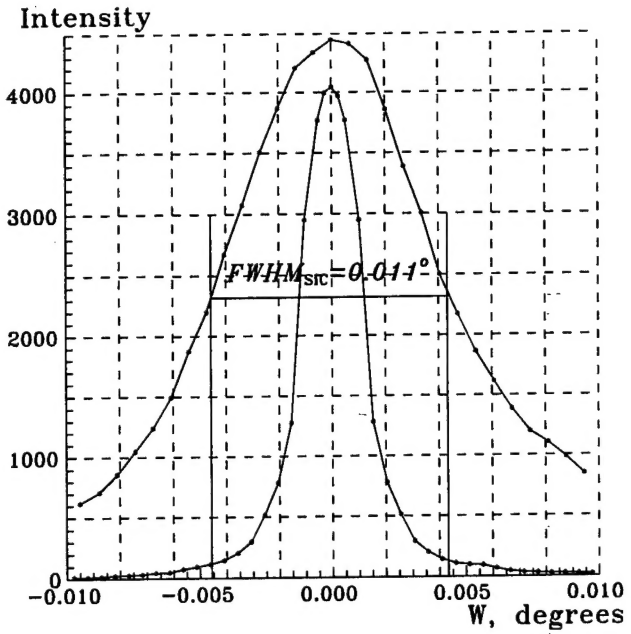

Fig.2. W–scanning of SiC substrate defect place reflex (0006), W_0=17.85°
FWHM=0.011° (=40arc.sec)
The same for polished and etched place
FWHM=0.0038°=14"

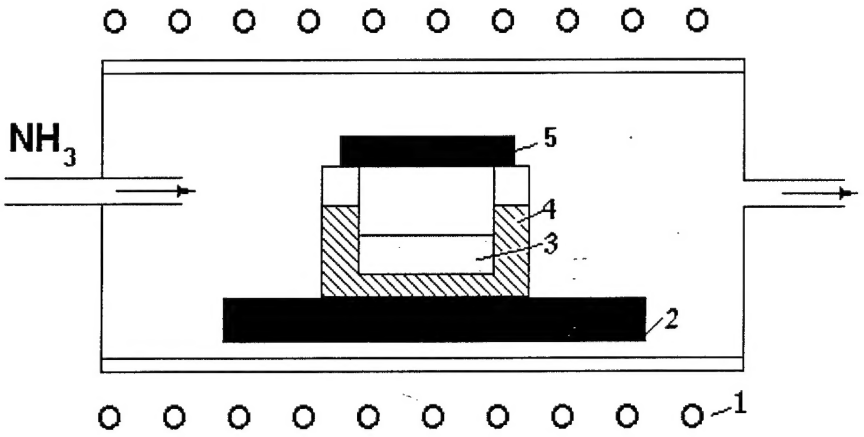

Fig.3. Experimental setup for growth of GaN layers by SSM

1.Inductor
2.Susceptor
3. Vapor source
4 Container
5. SiC-substrate

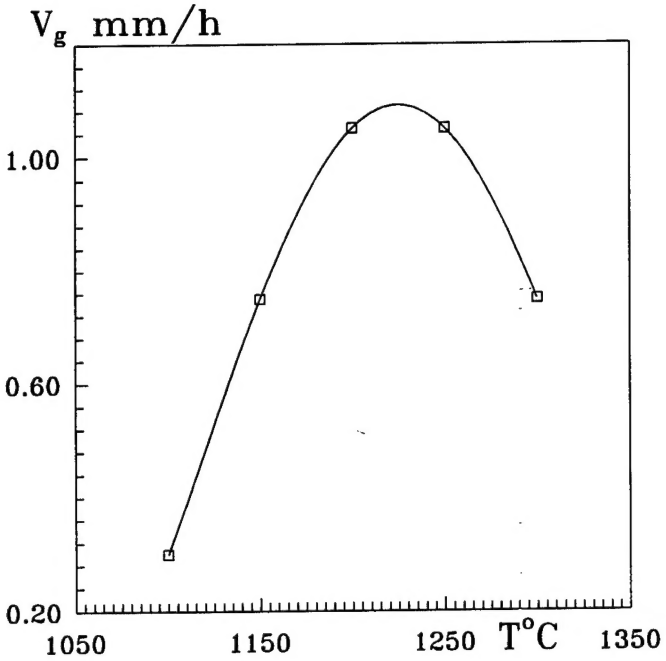

Fig.4 Dependence of GaN layers growth rate on temperature

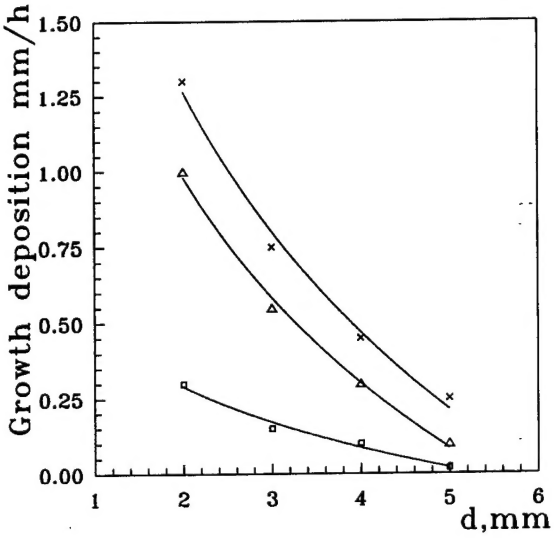

Fig.5. Dependence of GaN growth rate on distance between the substrate and source.
Growth temperature – 1200°C
Input ammonia flow – 40l/h.